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AN X-RAY STUDY OF FIREBRICK

BY

ALBERT E. R. WESTMAN

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AN X-RAY STUDY OF FIREBRICK

I. Introduction

1. *Preliminary Statement.*—When a thin section or a powdered sample of a commercial firebrick is examined under a petrographic microscope, a considerable portion of the material is found to be so finely divided or so deeply colored that its mineralogic composition cannot be determined. This is particularly true of soft-fired bricks or bricks containing relatively large percentages of iron or titanium. Even with the best microscopic equipment, it is difficult to make positive identification of crystals in a firebrick whose diameters are smaller than about 100 000 Angstrom units.* With X-rays, on the other hand, it is possible to obtain diffraction patterns from particles with diameters as small as about 50 Angstrom units, or about one two-thousandth the diameter of the smallest particles suitable for petrographic work. It was the purpose of the investigation reported in this bulletin to obtain powder diffraction patterns of a number of brands of commercial firebricks, to find what information could be obtained from the patterns, and to compare the information, so obtained, with that secured by thermal expansion measurements, petrographic examinations, and chemical analyses.

A summary of previous investigations in which X-rays were used in studying ceramic materials is given in Table 1. It will be observed that practically no X-ray studies of fireclay bricks have been made, although some relatively pure ceramic materials have been studied.

Samples of a number of brands of firebrick had been obtained in connection with investigations of checkerbrick and their physical and chemical properties had been determined. These samples were used in this investigation. The X-ray powder diffraction patterns of fourteen brands were obtained, twelve of the brands being commercial fireclay bricks, and two being commercial firebricks which would ordinarily be considered special refractories. The pattern of a sample of pure quartz was also obtained. The patterns of cristobalite, mullite, and calcined alumina had been obtained by McVay and Thompson† with the same apparatus and methods. In all cases the spacings of the lines in the patterns were determined.

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*An Angstrom unit (Ang. or A. U.) is 10^{-8} cm., i.e., one one-hundred millionth of a centimeter.

†McVay, T. N. and Thompson, C. L., "An X-ray Investigation of the Effect of Heat on China Clays," Jour. Am. Cer. Soc., 11 (11), pp. 829-41, 1928.

TABLE 1

WORK OF PREVIOUS INVESTIGATORS

Investigator	Materials	Data Reported	Method
(1) Louis Navias	Three ball clays, china clay, mullite.	None	Hull powder method
(2) Navias and Davey	Mullite and natural sillimanite.	Patterns and spacings	do.
(3) J. T. Norton	Cyanite and andalusite.	do.	do.
(4) J. T. Norton	Mech. mix. of alumina and silica in mol. proportion 1:1, and 3:2, as well as kaolin. All heated above 1500° C.	do.	do.
(5) A. Hadding	Fired china clay, fireclay, quartz, kaolin and fused clay.	Patterns	Debye-Scherrer powder method
(6) Bragg, Shearer, and Mellor	Two porcelain clays, 4 specimens of kaolin, 2 spec. sillimanite. Heated to temp. from 100° C–1000° C.	Patterns	Debye-Scherrer powder method
(7) G. Shearer	China clay, china clay and alumina, and alumina heated to temp. from 1200° C–1700° C. Also sillimanite.	Spacings	do.
(8) J. F. Hyslop	China clay, German and Eng. siliceous clay, pl. Ger. clay, Eng. ball clay. Heated to temp. from 450° C–1450° C.	Patterns	Diffraction pattern from flat surface
(9) A. Hadding	Quartz, kaolin, mica.	Spacings	Debye-Scherrer powder method
(10) F. Rinne	Zettlitz kaolin and nacrite from Freiberg. Bauxite, diaspore, silica, corundum.	Spacings, patterns	Powder method
(11) O. Krause	Quartz, kaolin, sillimanite, and porcelain.	Angles of incidence	Debye-Scherrer powder method
(12) F. Rinne	Products of calcination of calcite, dolomite, and kaolinite.	Spacings	Powder method
(13) F. Rinne	Kaolin, sillimanite, mullite, tridymite, cristobalite and dolomite.	Pattern tracings	Powder method

1. L. Navias, "Quantitative Determination of the Development of Mullite in Fired Clays by an X-ray Method," J.A.C.S., 8, 296, 1925.
2. Navias and Davey, "Difference between Sillimanite and Mullite by Their X-ray Diffraction Pattern," J.A.C.S., 8, 640, 1925.
3. J. T. Norton, "An X-ray Study of Cyanite and Andalusite," J.A.C.S., 8, 636. 1925.
4. J. T. Norton, "An X-ray Study of Natural and Artificial Sillimanite," J.A.C.S., 8, 401, 1925.
5. A. Hadding, "X-ray Investigation of Clays and Other Ceramic Substances," Trans. Eng. Cer. Soc. 24, 27, 1924-25.
6. Bragg, Shearer, Mellor, "Notes on Crystalline Structure of Certain Clays and China Clays by the X-ray Powder Method," Trans. Eng. Cer. Soc., 22, 105, 1922-23.
7. G. Shearer, "Results of an X-ray Investigation of the Crystalline Nature of China Clays," Trans. Eng. Cer. Soc., 23, 314, 1923-24.
8. J. F. Hyslop, "The Constitutional Changes Occurring in Clays on Heating," Trans. Eng. Cer. Soc., 24, 402, 1924-25.
9. A. Hadding, "Eine Röntgenographische Methode Kristalline und Kryptokristalline Substanzen zur Identifizieren," Zeit. Kryst., 58, 108, 1923.
10. F. Rinne, "Röntgenographische Untersuchungen an Einigen Feinzerteilten Mineralien, Kuntstprodukten, und Dichten Gesteinen," Zeit. Kryst., 60, 55, 1924.
11. Dr. O. Krause, "Andwendung von Röntgenographie und Fluoreszenz-strahlung in der Feinkeramik," Ber. Deut. Ker. Gesel., 8, 2, 114, 1927.
12. F. Rinne, Zeit. Kryst., 60, 63, 1925.
13. F. Rinne, "The Use of X-rays in Ceramics," Ker, Rund., 33, 427-9, 459-60, 525-7, 1925.

TABLE 2

DATA PERTAINING TO MANUFACTURE

Brand	Location of Plant	Method of Manufacture*	Burning Temperatures	Raw Materials
I	California	H.M.	17–18	lean, "bone" clay, like bauxite
II	Missouri	D.P.	7†	2nd. grade semi-flint and plastic clays
V	Illinois	S.M.R.	9–11	plastic clay
VI	Pennsylvania	H.M.	10–12	flint and plastic clays
VII	Missouri	S.M.R.	8	plastic clay, flint clay and grog
IX	Kentucky	H.M.	11	flint and semi-flint clay
XI	New Jersey	H.M.	12	plastic clays
XIII	Kentucky	S.D.P.	8	flint, semi-flint, and plastic clays
XV	Missouri	D.P.	7	flint, and semi-flint clay
XVI	New Jersey	S.M.R.	15‡	calcined raw material and plastic clay
XVII	Missouri	S.M.R.	10	high grade plastic clay
XVIII	Ohio	A–B	31	Ga. kaolin, flint, semi-flint and plastic clays
XIX	Illinois	S.M.R.	11–12	Mo. flint and brand V plastic clay
XXIII	Missouri	H.M.	14	Mo. diaspore, high grade plastic clay

*S.M.R., stiff-mud repress; D.P., dry press; S.D.P., semi-dry repress; A–B, auto-brick; H.M., hand-made.
†Reported as 2200–2400° F.
‡Reported as 2600° F.

2. *Data Previously Secured.*—Information regarding the method of manufacture, raw materials, etc., of the brands investigated is given in Table 2. It will be seen that a rather wide range of materials was included. The chemical analyses of the different brands, as reported by the manufacturers, are given in Table 3. The approximate mineralogical composition of the different brands had been determined (1) by calculations based on the thermal expansion curves, (2) by petrographic examination using the powder immersion method, and (3) by calculations based on the chemical analyses. A detailed description of these methods and the data obtained are given in Bulletin 181.* The more pertinent part of the data is reproduced in Chapter IV of this bulletin.

3. *Acknowledgments.*—The writer is indebted to PROF. C. W. PARMELEE, Head of the Department of Ceramic Engineering, for his interest and assistance in providing facilities for this investigation; also to DR. G. L. CLARK of the Chemistry Department, who kindly permitted the use of the X-ray equipment.

This investigation has been carried on as a part of the work of the Engineering Experiment Station of the University of Illinois, of

*"The Thermal Expansion of Fireclay Bricks," Univ. of Ill. Eng. Exp. Sta. Bul. 181, 1928.

TABLE 3

ANALYSES OF FIRECLAY BRICKS

Brand	SiO_2	Al_2O_3	Fe_2O_3	TiO_2	CaO	MgO	Alkalies	Total
	Per Cent							
I	51.00	44.00	2.1	1.1	←———	1.8	———→	100.00
II	60.40	34.04	2.28	1.84	0.59	0.54	1.19	100.88
V	64.60	30.13	2.73	1.52	0.44	0.45	0.63	100.50
VI	59.20	35.60	2.60	....	0.40	0.40	1.80	100.00
VII	52.53	41.12	2.56	1.64	0.23	1.05	0.87	100.00
IX	53.32	41.51	1.17	2.46	0.28	0.44	0.66	99.84
XI	63.60	31.38	....	....	....	....	.....	.....
XIII	54.10	41.20	2.02	1.83	0.30	0.29	0.70	100.44
XV	59.1	36.0	1.1	2.8	....	....	0.8	99.80
XVI	73.46	22.08	1.50	1.94	0.14	0.20	0.58	99.90
XVII	59.76	35.90	2.63	0	0.22	0.64	0.84	99.99
XVIII	51.96	45.38	0.57	1.73	0.10	0.22	($0.04SO_3$)	100.00
XIX	53.78	40.76	2.12	1.80	0.52	0.28	0.95	100.21
XXIII	19.66	75.03	1.01	3.40	0.21	0.38	0.49	100.18

which DEAN M. S. KETCHUM is the director, and of the Department of Ceramic Engineering of which PROF. C. W. PARMELEE is the head.

II. POWDER DIFFRACTION MEASUREMENTS

4. *Preparation of Specimens.*—For each brand of brick a number of fragments of about 30 gm. total weight were broken from a brick and crushed to pass a 200-mesh sieve. The portion of this powder which passed through a 300-mesh sieve was used in preparing a specimen. The quartz sample was prepared by water-grinding in an agate mortar. A small plug of absorbent cotton was inserted in a thin-walled glass tube about 5 cm. long, the plug being pushed to a central position. The tube was filled for a distance of about 1.5 cm. on one side of the plug with the 300-mesh powder of the brick under examination and for the same distance on the other side of the plug with 300-mesh c.p. sodium chloride, the tube was rubbed with a file to obtain close packing and then cotton plugs were inserted at each end to keep the powders in position.

5. *Apparatus and Method.*—The apparatus was one developed by the General Electric Company and was essentially the same as that described by Davey,* the casette arrangement adapted to the use of

*Davey, W. P., "A Study of Crystal Structure and Its Applications," Gen. Elec. Rev. 28, p. 286, 1925.

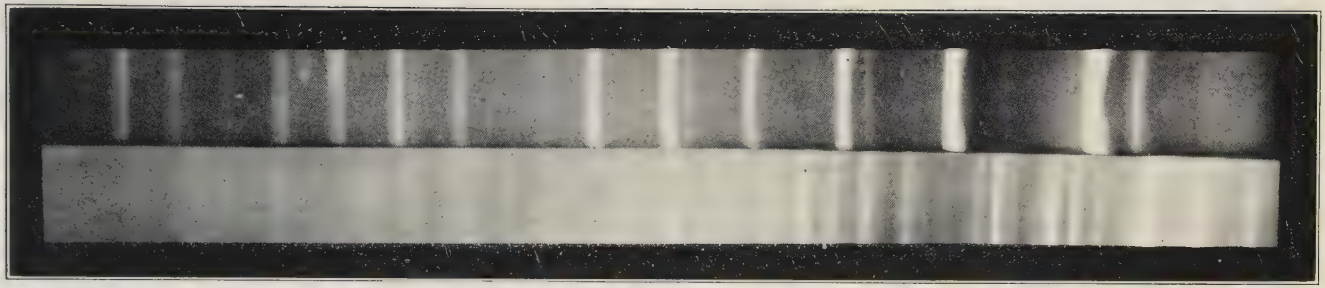

FIG. 1. TYPICAL POWDER DIFFRACTION PATTERN OF A FIRECLAY BRICK (BRAND VI)

Upper band is a sodium chloride pattern.
Lower band is a firebrick pattern.

the Hull* powder method being used. A beam of X-rays from a Coolidge tube with a molybdenum target passed through a narrow slit and then through the specimen prepared as described in the foregoing section. The undiffracted and diffracted rays passed through a zirconia filter and registered on a photographic film 40 cm. long and 4.8 cm. wide, which was bent so as to form the arc of a quadrant having the specimen at its center. Practically all the rays except those having wave lengths corresponding to the $K\alpha$ doublet of molybdenum were absorbed in the filter, so the effects of a monochromatic beam were secured. On developing a film, two diffraction patterns were obtained, one for the sodium chloride and the other for the firebrick. Each pattern consisted of a series of parallel lines of varying intensities and spacings, the print from a typical negative (from Brand VI) being shown in Fig. 1.

6. *Operation.*—For most of the brands exposures of 48 hours were made, but in one or two cases longer exposures were necessary, the longest being 72 hours. It was found that the films from exposures of 48 hours contained as many lines as and were easier to measure than those exposed for a longer period.

7. *Determination of Spacings.*—The angle through which the beam of X-rays was diffracted by the powders depended on the spacings of the parallel planes in the crystal lattices of the compounds present. These spacings were determined for the lines in the diffraction patterns by the use of a measuring device provided with a special scale which was supplied with the apparatus. In this way the spacings were read directly in Angstrom units. The lines for the sodium chloride were used as calibrating lines, the theoretical values used as standard being those based on a unit cell dimension of 2.814 Angstrom units for sodium chloride. For each film the sodium chloride line corresponding to a theoretical spacing of 1.990 Angstrom units was used in setting the film in the measuring device. Readings were made at the centers of the lines or, where the $K\alpha_1$ and $K\alpha_2$ lines were apparently resolved, half way between the lines of the doublet. The readings for both the sodium chloride and the firebrick were made three times and average values were calculated and then corrected for differences between the observed sodium chloride spacings and the theoretical values. These corrections were seldom greater than 0.0002 Angstrom units.

The values obtained are given in Tables 4, 5, and 6. Table 4 gives the spacings of the fireclay bricks, Table 5 those of Brand XVIII,

*Hull, A. W., "A New Method of X-ray Crystal Analysis," Phys. Rev., 10, p. 661, 1917.

which was a kaolin brick, and of mullite, cristobalite, and quartz, Table 6 those of Brand XXIII, which was largely diaspore and calcined alumina. For convenience of reference the lines have been numbered in order. The line numbers of Tables 4 and 5 correspond, those of Table 6 are different. The spacings of the different lines found in the fireclay brick patterns were averaged, the averages being reported in Tables 4 and 5. The average relative intensities of the lines are shown in Table 4, S meaning strong, M, medium, F, faint, and VF, very faint. All the fireclay bricks gave approximately similar patterns.

8. *Comparison of Patterns.*—The 55 lines which were found for fireclay bricks, the spacings of which are reported in Table 4, can be attributed to the presence of mullite, cristobalite, or quartz (with a few exceptions) by the comparison afforded in Table 5. Thus, lines 14, 25, 26, 32, 33, and 42 could be attributed to mullite, cristobalite, or quartz; lines 7, 17, 19, 21, 23, 28, and 37 could be attributed to mullite or cristobalite; lines 5, 10, 12, 13, 18, 35, 41, 43, 47, 52, and 53 to either mullite or quartz; lines 16, 27, 29, and 34 to cristobalite or quartz; lines 3, 6, 15, and 24 to cristobalite; lines 2, 30, 38, 39, and 46 to quartz; and lines 1, 4, 8, 9, 31, 36, 44, 45, 49, 50, and 54 to mullite. In addition, the combination of lines 9 and 10 might correspond to one line reported for either cristobalite or quartz; lines 15 and 16 to one line reported for mullite, lines 19 and 20 for quartz, lines 22 and 23 for quartz, lines 29 and 30 for mullite and lines 38 and 39 for mullite. Lines 11, 40, 48, 51, and 55 are then left. Lines 48, 51, and 55 could probably be attributed to mullite, cristobalite, or quartz if their patterns had been a little denser. Lines 11 and 40 were found in only one brand each; they might possibly be due to other minerals. Line 3 is the most useful line in deciding whether cristobalite is present, as it is a strong line and is not close to other lines. Line 6 is also specific for cristobalite but is not strong or persistent. A number of lines can be used for showing the presence of mullite. In the case of quartz, however, characteristic lines are scarce. Lines 2, 30, 38, 39, and 46 might be definite indications of quartz in the presence of mullite and cristobalite but line 30 is probably due to mullite, line 46 is seldom found, line 2 is specific but not persistent, and lines 38 and 39 might be combined and explained as mullite, although that is probably not their true explanation.

9. *Conclusions.*—By using the relations given in the last section the following conclusions can be drawn: (1) All the fireclay bricks

TABLE

SPACINGS OF X-RAY LINES OF FIREC

Brand	Line Numbers (a																											
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	
I	5.3	....	4.08	....	3.40	....	2.88	2.69	2.52	2.42	.....	2.28	2.21	2.12	2.035	.....	1.882	1.830	1.701	1.630	1.601	.....	1.525	.....	1.444	1.404	1.370	1.
II	5.3	....	4.06	....	3.36	3.14	2.87	2.68	2.51	2.41	.....	2.28	2.20	2.12	2.03	1.938	1.876	1.824	1.691	1.624	1.593	.....	1.522	.....	1.442	.....	1.381	1.
V	5.35	....	4.06	....	3.37	3.14	2.86	2.68	2.52	2.42	.....	2.28	2.204	2.123	2.034	1.944	1.880	1.831	1.699	1.618	1.598	.....	1.526	.....	1.439	1.401	1.371	1.
VI	5.38	4.25	4.05	....	3.36	....	2.872	2.677	2.527	2.422	2.375	2.283	2.205	2.120	.....	1.960	1.880	1.825	1.688	.....	1.596	.....	1.522	1.475	1.443	1.399	1.373	1.
VII	5.35	....	4.08	3.74	3.39	3.16	2.88	2.695	2.525	2.42	.....	2.28	2.208	2.122	2.04	1.955	1.883	1.836	1.701	1.628	1.602	.....	1.526	.....	1.443	1.404	1.368	1.
IX	5.38	....	4.10	....	3.38	....	2.87	2.70	2.54	2.428	.....	2.29	2.217	2.125	2.036	1.96	1.888	1.838	1.70	1.632	1.600	.....	1.524	.....	1.446	1.403	1.372	1.
XI	5.38	4.26	4.08	....	3.36	3.17	2.875	2.70	2.52	.....	.....	2.282	2.217	2.125	2.038	.....	1.880	1.823	1.702	1.612	.....	.....	1.531	.....	1.443	.....	1.373	1.
XIII	5.37	....	4.07	3.78	3.38	3.16	2.868	2.680	2.532	2.420	.....	2.280	2.218	2.120	2.038	1.962	1.883	1.830	1.697	1.627	1.601	1.573	1.525	.....	1.441	1.401	1.369	1.
XV	5.30	....	4.06	....	3.36	....	2.86	2.68	2.52	2.42	.....	2.295	2.21	2.12	2.03	.....	1.879	1.821	1.692	1.617	1.595	.....	1.521	.....	1.440	1.395	1.372	1.
XVI	5.39	....	4.08	....	3.38	3.17	2.872	2.695	2.52	.....	.....	2.292	2.219	2.125	2.035	1.955	1.884	1.822	1.701	1.616	.....	.....	1.535	.....	1.446	.....	1.377	1.
XVII	5.3	....	4.06	....	3.36	....	2.86	2.69	2.52	2.43	.....	2.28	2.20	2.125	2.035	.....	1.878	1.821	1.691	1.624	1.596	.....	1.525	.....	1.440	.....	1.369	1.
XIX	5.38	....	4.05	3.76	3.39	3.16	2.872	2.68	2.523	.....	.....	2.278	2.215	2.130	2.040	1.958	1.883	1.828	1.701	1.633	1.607	.....	1.531	.....	1.445	1.404	1.374	1.
Average	5.35	4.26	4.07	3.76	3.37	3.16	2.870	2.687	2.523	2.421	2.375	2.283	2.210	2.123	2.036	1.954	1.881	1.827	1.697	1.624	1.599	1.573	1.526	1.475	1.443	1.401	1.372	1.
Rel. Intens.	M	F	S	M	S	VF	F	F	M	VF	F	F	M	F	VF	VF	VF	VF	F	VF	VF	VF	M	VF	F	VF	VF	V

TABLE 5

SPACINGS OF X-RAY LINES OF SPECIAL

Material	Line numbers (arb																											
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28
Av. F.C. Brick	5.35	4.26	4.07	3.76	3.37	3.16	2.870	2.687	2.523	2.421	2.375	2.283	2.210	2.123	2.036	1.954	1.881	1.827	1.697	1.624	.....	1.573	1.526	1.475	1.443	1.401	1.372	1.3
Br. XVIII	5.30	....	....	....	3.37	....	2.870	2.673	2.533	2.417	.....	2.279	2.203	.....	.....	.....	1.878	.....	1.698	.....	1.599	1.571	1.521	.....	1.441	1.397	.....	1.3
Mullite	5.30	....	....	3.75	3.39	....	2.85	2.69	2.53	2.41	.....	2.28	2.19	2.11	2.000	.....	1.885	1.833	1.699	.....	1.598	.....	1.523	.....	1.445	1.404	.....	1.3
Cristobalite	....	....	4.03	....	....	3.12	2.83	.....	.....	2.47	.....	.....	.....	2.11	2.01	1.93	1.87	.....	1.69	.....	1.60	.....	1.53	1.492	1.430	1.395	1.360	1.3
Quartz	....	4.25	....	....	3.35	....	.....	.....	.....	2.45	.....	2.277	2.223	2.125	.....	1.977	.....	1.817	1.667	.....	.....	.....	1.542	.....	1.448	1.406	1.371	...

TABLE

SPACINGS OF X-RAY LINES OF SPECIAL

Material	Line numbers (																											
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	2
r. XXIII	5.30	3.45	3.36	3.21	2.85	2.67	2.52	2.365	2.28	2.195	2.07	1.828	1.789	1.734	1.683	1.594	1.515	1.434	1.396	1.367	1.328	1.272	1.232	1.443	1.221	1.098	1.076	1
ullite	5.30	....	3.37	(VF)	2.87	2.67	2.53	.....	2.28	2.203	....	1.833	.....	.....	1.699	1.598	1.523	1.445	1.404	.....	1.332	1.267	1.235	.....	1.218	1.100	1.06	..
$_2O_3$	....	3.47	....	....	....	....	2.55	2.42	2.31	2.11	1.98	1.80	.....	.....	.....	1.59	.....	1.43	.....	1.39	1.30	1.26	1.23	.....	.....	.....	.....	..

Brick (in Angstrom units)

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29	30	31	32	33	34	35	36	37	38	39	40	41	42	43	44	45	46	47	48	49	50	51	52	53	54	55
1.279	1.262	.....	.....	1.189	.....	.....	.....	1.097	.....	.....	.....	1.006	0.986	.....	0.952	0.926	.....	0.895	.....	.....	0.850	.....	.....	.....	.....	.....
1.277	1.258	.....	.....	1.191	.....	.....	1.121	1.101	1.080	1.045	.....	1.009	0.985	.....	0.950	0.923	.....	0.895	0.875	.....	0.851	.....	.....	0.812	0.798	.....
1.278	.....	.....	.....	1.191	.....	.....	.....	1.101	.....	.....	.....	1.010	0.993	.....	0.947	0.923	.....	.....	.....	.....	0.848	0.833	.....	0.815	.....	.....
1.275	1.258	1.237	.....	1.189	.....	.....	.....	1.101	1.077	1.042	.....	1.011	0.986	.....	0.949	0.920	.....	0.893	0.875	0.860	0.847	0.837	.....	.....	.....	.....
1.279	1.261	1.239	1.217	1.191	.....	.....	1.121	1.101	.....	.....	.....	1.008	0.992	.....	0.947	0.925	.....	0.897	.....	.....	0.850	.....	0.830	.....	.....	.....
.....	1.266	.....	.....	1.188	.....	1.147	1.121	1.099	.....	.....	.....	1.013	0.990	.....	0.943	.....	.....	.....	.....	0.857	0.846	.....	.....	.....	.....	.....
1.281	1.260	.....	1.219	1.195	1.180	1.149	.....	1.101	1.080	1.047	1.035	1.012	0.989	.....	0.943	0.926	0.913	0.891	.....	.....	0.848	.....	0.826	0.813	.....	.....
1.277	1.260	1.238	1.217	1.189	.....	1.149	1.122	1.100	.....	.....	.....	1.013	0.988	.....	0.947	0.922	.....	0.893	0.874	.....	0.848	.....	0.826	0.812	.....	.....
1.277	1.258	.....	.....	1.193	.....	.....	.....	1.101	.....	.....	.....	1.008	0.985	.....	0.947	0.920	.....	0.896	.....	.....	.....	.....	0.830	0.817	0.795	.....
1.282	1.260	1.237	.....	1.188	.....	1.154	.....	1.104	1.082	1.041	.....	1.010	0.988	0.962	0.943	0.917	.....	0.894	0.872	.....	0.850	.....	0.828	0.812	0.791	.....
1.274	1.258	.....	.....	1.187	.....	.....	.....	1.097	.....	.....	.....	1.005	.....	.....	0.952	0.923	.....	0.895	.....	.....	0.854	.....	0.828	.....	.....	.....
1.271	.....	1.241	1.215	1.189	.....	1.154	.....	1.102	1.081	1.042	.....	1.013	0.992	.....	0.947	.....	.....	0.894	.....	.....	0.849	.....	.....	0.812	.....	0.771
1.277	1.260	1.238	1.217	1.190	1.180	1.151	1.121	1.100	1.080	1.043	1.035	1.010	0.989	0.962	0.947	0.922	0.913	0.894	0.874	0.858	0.849	0.835	0.828	0.813	0.795	0.771
VF	VF	VF	VF	VF	VF	VF	VF	VF	VF	VF	VF	VF	VF	VF	VF	VF	VF	VF	VF	VF	VF	VF	VF	VF	VF	VF

terials (in Angstrom units)

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29	30	31	32	33	34	35	36	37	38	39	40	41	42	43	44	45	46	47	48	49	50	51	52	53	54	5
.277	1.260	1.238	1.217	1.190	1.180	1.151	1.121	1.100	1.080	1.043	1.035	1.010	0.989	0.962	0.947	0.922	0.913	0.894	0.874	0.858	0.849	0.835	0.828	0.813	0.795	0.771
.275	1.260	1.240	1.216	1.191	.....	.....	1.121	1.100	.....	.....	.....	1.012	0.988	....	0.947	0.925	.....	0.896	.....	0.860	0.848	.....	0.826	0.812	0.795	.....
.267	.....	1.235	1.218	1.190	.....	1.152	1.120	1.100	1.06	.....	.....	1.008	0.988	0.963	0.947	0.925	.....	0.895	.....	0.860	0.849	.....	0.826	0.812	0.796	.....
.275	.....	.....	1.225	1.205	1.170	.....	.....	1.095	.....	.....	.....	.....	0.987	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
.285	1.254	.....	1.226	1.201	1.182	1.153	.....	.....	1.082	1.042	.....	1.015	0.986	0.959	.....	.....	0.914	0.895	.....	.....	.....	.....	0.827	0.809	.....	.....

terials (in Angstrom units)

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29	30	31	32	33	34	35	36	37	38	39	40	41	42	43	44	45	46	47	48	49	50	51	52	53	54	55
996	0.905	0.856	0.830	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
...	.....	.....	0.826	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
...	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....

gave very definite and pronounced mullite lines and, in fact, gave patterns which resembled each other very closely. (2) All the fireclay bricks gave cristobalite lines, line 3 occurring in all patterns and line 6 in Brands II, V, VII, XI, XIII, XVI, and XIX, which might be taken to indicate the presence of relatively large amounts of cristobalite in these brands. (3) Faint lines which might definitely be attributed to quartz were found for Brands II, VI, XI, XVI, and XIX, which would indicate that they were relatively high in quartz content. (4) Brand XVIII, which was made from kaolin, gave mullite lines only. (5) The pattern of Brand XXIII, which was made largely from diaspore, was quite different from those of the fireclay bricks. As shown in Table 6 many of the lines of Brand XXIII could be attributed to mullite or alumina although the pattern could not be explained as satisfactorily as that of the fireclay bricks.

III. Comparison with Data Obtained by Other Methods

10. *Previous Data.*—Table 7 gives a summary* of the information regarding the mineralogic composition of the brands investigated which was obtained by thermal expansion measurements, petrographic examinations, and chemical analyses. While the agreement between the values obtained by different methods is only approx-

Table 7
Mineralogic Composition of Bricks

Brand	Calculated from Thermal Expansion Data			Calculated from Petrographic Data	Calculated from Chemical Analyses
	Cristobalite Vol. per cent	Quartz Vol. per cent	Free Silica Vol. per cent	Quartz Vol. per cent	Free Silica Wt. per cent
I	12.2	3.1	15.3	10.2	(0.7 excess Al_2O_3)
II	11.4	5.2	16.7	4.4	20.3
V	22.7	11.0	33.6	12.4	29.1
VI	1.0	6.2	7.2	6.5	17.3
VII	8.8	0.7	9.5	0.6	4.1
IX	4.6	3.1	7.7	4.5	4.4
XI	11.8	11.0	22.7	25.9	26.6
XIII	4.8	1.0	5.8	0.6	5.6
XV	7.2	6.2	13.4	15.0	16.7
XVI	24.7	23.8	48.6	33.0	47.5
XVII	8.7	6.7	15.3	7.2	17.5
XVIII	0	0	0	0	(1.3 excess Al_2O_3)
XIX	12.8	10.0	22.8	12.4	5.7
XXIII	0	0	0	0	(58.4 excess Al_2O_3)

*"The Thermal Expansion of Fireclay Bricks," Univ. of Ill. Eng. Exp. Sta. Bul. 181, 1928, p. 18 et. seq.

imate, yet there is sufficient agreement to indicate that (1) Brands I, II, V, XI, XVI, and XIX were high in cristobalite, (2) Brands V, XI, XVI, and XIX were high in quartz, and (3) Brands XVIII and XXIII contained practically no cristobalite or quartz.

11. *Comparison with X-ray Data.*—A comparison of these conclusions with those given in Chapter II shows agreement regarding high cristobalite content for Brands II, V, XI, XVI, and XIX, partial agreement for Brand VII and disagreement for Brands I and XII. For high quartz content the methods agree on Brands XI, XVI, and XIX but disagree to some extent on Brands II and VI and very decidedly on Brand V. The X-ray patterns of Brands XVIII and XXIII do not show characteristic quartz or cristobalite lines, indicating that these minerals are not present. This agrees with the data given in Table 7. Line 3, a strong cristobalite line, was present in all the fireclay patterns although the cristobalite content of some of the brands was low, that for Brand VI being approximately 1 per cent. Line 3 is, therefore, a sensitive criterion for the presence of cristobalite.

IV. Summary of Conclusions

12. *Summary of Conclusions.*—X-ray powder diffraction measurements of fourteen brands of firebrick have been described and the data obtained reported and analysed. A comparison with data obtained from thermal expansion, petrographic, and chemical studies has been made.

The following general conclusions may be drawn:

(1) The X-ray diffraction patterns of all the fireclay bricks were similar in appearance. Mullite lines and one cristobalite line were strongly developed in all the patterns. Nearly all quartz lines were masked by mullite and cristobalite lines and those not masked were rather faint.

(2) A brand of brick made from kaolin gave practically a mullite pattern, no cristobalite or quartz lines being present.

(3) A brand of brick made largely from diaspore gave a pattern which was quite different from fireclay patterns and apparently contained some mullite lines and alumina lines. The pattern could not be accounted for very satisfactorily on the assumption that only these substances were present.

(4) Practically all the lines in the patterns for fireclay and kaolin bricks could be attributed to mullite, cristobalite, or

quartz. There was no reason for believing that the particles in these bricks whose diameter was less than 100 000 Angstrom units consisted of minerals which were different from those whose diameter exceeded this and which could, therefore, be identified by petrographic methods.

(5) Estimates of the amounts of cristobalite and quartz present in the brands based on a comparison of their diffraction patterns agreed roughly with those based on thermal expansion and petrographic data. However, it was possible to make only very inaccurate estimates in the case of one or two brands.

Since mullite, cristobalite, and quartz produce patterns which overlap to a considerable extent, the development of X-ray apparatus which would give wider separation of the lines, and permit accurate intensity measurements, would be a boon to research on ceramic materials which nearly always contain these minerals.

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